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SPECIAL FEATURES

Fabrication, characterization and functionality control on the nanoscale

Challenges to novel science and technology to establish a new world of nanoelectronics

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Progress in semiconductor electronics, which began with the invention of the point-contact solid-state transistor in 1947, has been achieved by ceaseless efforts of miniaturization, integration, and improvement of performance of silicon-based semi- conductor electronic devices. However, it is widely believed that this progress will confront inevitable physical limits involved in the traditional silicon-based semiconductor devices in the near future. To overcome the limits and establish a more advanced world of electronics, or nanoelectronics, vigorous research and development are now underway by utilizing various advanced techniques developed in nanotechnology around the world, particularly in Japan, the United States, and Europe. This trend is summarized in the accompanying figure.

What are the key technologies for the development of nanoelectronics? We believe that it is essential to develop revolutionary methods for fabricating nanomaterials and nano- structures at will, measuring their physical properties directly, and controlling their functions according to predetermined design.

The present issue reports new advances in each of the key technologies mentioned above, which have been achieved in NIMS recently. The first two articles report that a single conductive polymer nanowire can be formed at a designated position by inducing local chain polymerization using a probe tip of the scanning tunneling microscope and that a conductive tungsten oxide nanorod with a diameter of several nanometers can be formed at a chemically active position using chemical vapor deposition. Because nanowires and nanorods are useful for electrical wiring of integrated nanoelectronic devices, we can think of a variety of applications of the new methods for the development of nanolectronics.

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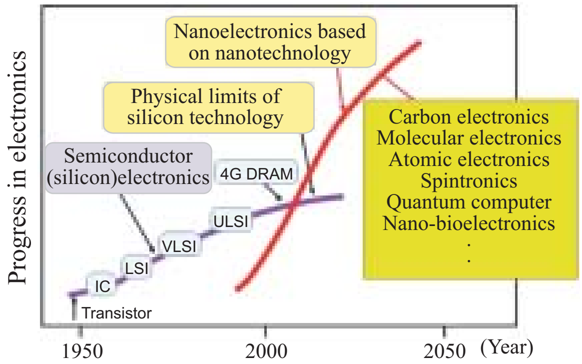
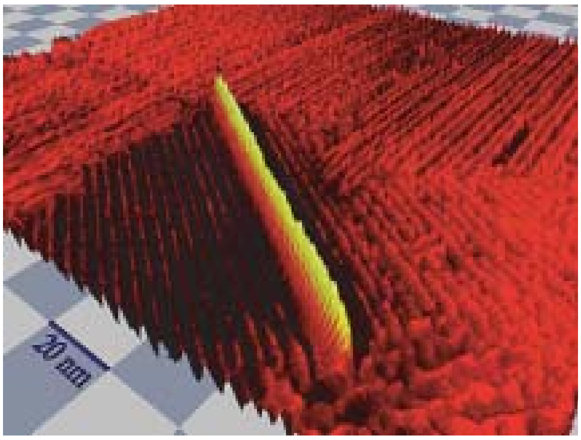


Fig. History and future outlook for electronics.



Reference Fig. Conductive nanowire fabricated by chain polymerization reaction (See story, p.2).

In this issue

NIMS NEWS

Second Swiss-Japanese Workshop on Biomaterials

< Continued on p.3

Fabrication of conductive polymer nanowire

—Nano-level control of chain polymerization reaction—

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With miniaturization of silicon devices now approaching its limits, development of nanometer (10^{-9}) size devices which operate on the basis of new concepts has become an urgent task. However, even assuming that individual nano-devices can be created, interconnection and integration of these devices is still an important problem. Thus, development of a nanowiring technology for transmitting electricity at nanometer widths is indispensable for realizing next-generation nanodevices.

The Atomic Electronics Group recently developed a technology for controlling the chain polymerization reaction of organic molecules with spatial resolution on the order of 1 nm, and succeeded in fabricating conductive polymer nanowires with a width of only one molecule (approx. 3 nm) in designated lengths and at designated positions (**cover, reference Fig.**).

The procedure is illustrated in **Fig. 1**. The organic molecule used is a diacetylene compound which contains two C≡C triple bonds. When placed on a graphite surface by an appropriate method, these molecules spontaneously form a molecular film with the molecules arranged in a regular pattern of straight lines. Next, a pulse voltage is applied to one arbitrarily selected molecule using the probe of a scanning tunneling microscope (STM). This triggers a one-dimensional chain polymerization reaction originating at that molecule, forming a polydiacetylene compound, which is a conductive polymer. As a result, a conductive nanowire with a one-molecule width can be fabricated nearly instantaneously, simply by applying a single stimulus at one arbitrary point.

Fig. 2 shows STM images of the material before and after the chain polymerization reaction. The bright line in **Fig. 2(b)** is the formed polymer nanowire.

This technique offers numerous advantages. For example, it enables fabrication of defect-free polymer nanowires with good linearity, and it is efficient, because it utilizes a spontaneous high speed process. Moreover, except for the initial stimulation, the reaction proceeds using only thermal energy. We are now engaged in both basic and applied research aimed at elucidation of the fundamental properties of nanowires, application as nanowiring in future nanodevices, and development of new nanodevices and sensors.

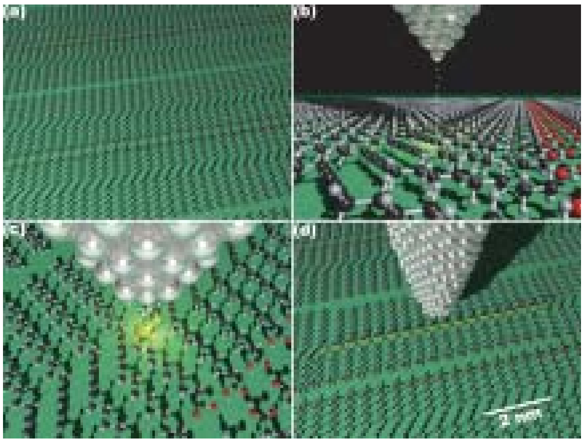

Fig. 1 Nanowire fabrication procedure: On the molecular film in (a), a stimulus is applied to an arbitrary molecule using an STM probe (b), causing excitation (c), which triggers a chain polymerization reaction, resulting in formation of a nanowire (yellow line) (d).

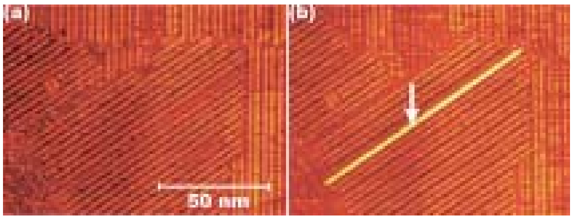

Fig. 2 (a) STM image of molecular film consisting of diacetylene compound, and (b) nanowire formed by applying stimulus with STM probe at position indicated by arrow.

Election of Nanomaterials Laboratory's Director-General Masakazu Aono as a Fellow of the American Vacuum Society

The Director-General of NIMS' Nanomaterials Laboratory(NML), Dr. Aono, has been elected as a Fellow of the American Vacuum Society (AVS). The award ceremony was held during the AVS 50th International Symposium in Baltimore on November 5. The citation for this award recognized Dr. Aono's achievements in research, mentioning "For pioneering work on the formation of nanostructures at surfaces and on surface analysis by low energy ion back-scattering." Dr. Aono is the third Japanese researcher to be named a Fellow of the AVS, following Prof. Chikara Hayashi (Senior Advisor, ULVAC, Inc.) in 1998 and Dr. Yasuhiro Horiike (former Prof., The University of Tokyo, currently NIMS Fellow).

Start of Joint Research with KIST for the 21st Century Frontier R&D Program, "Development of Nanostructure Material Technologies" by the Korean Ministry of Science and Technology

The Metallic Nanostructures Group (Director, Dr. Kazuhiro Hono) of the NIMS Materials Engineering Laboratory (MEL) is set to begin joint research with the Korea Institute of Science and Technology (KIST) on "Development of Nanostructure Material Technologies" under a commission from the Center for Nanostructured Materials and Technology (CNMT) (Director, Dr. Sang-Hee Suh) as part of the Nanostructure Material Technology Development program in the Korean Ministry of Science and Technology's 21st Century R&D Program. Research on atomic level analysis of the nanostructure of metallic nanostructure materials will be carried out jointly with Dr. Kyung-Tae Hong of KIST. To accomplish the goals of this research, NIMS will receive research funding and doctoral-level researchers dispatched from KIST, and will analyze the nanostructures of metallic nanomaterials developed in the above-mentioned program using its 3-dimensional atom probe. This work is expected to establish the cause-and-effect relationship between the nanostructure and mechanical properties of nano-materials and contribute thereby the development of high strength, high toughness, and high performance materials.

Fabrication and application of conductive metal oxide nanorods

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Recent years have seen the development of technologies for fabricating interesting nanostructures by a variety of methods. The Electro-nanocharacterization Group developed a multiple-probe scanning tunneling microscope (MP-STM) which enables direct measurement of the physical properties of these nanostructures, and in actual measurements, has shown that the MP-STM is an extremely powerful tool for investigating the functions of nanostructures. The key to further improvement in MP-STM capacity is completion of a fabrication technology for extremely thin probes.

In an MP-STM, multiple probes are placed in contact with a single nanostructure. This means that smaller nanostructures can be investigated as the spacing between the probes is reduced. Probe spacing is determined by the curvature radius of the probe tip and is on the order of 100 nm in ordinary probes. In contrast to this, we developed a new, extremely thin probe which realizes a probe spacing of less than 10 nm by fabricating a nanomaterial with a long, thin shape on the tip of an ordinary probe.

In previous examples, carbon nanotubes have been used as a nanomaterial on probe tips, but various problems remained, including control of electrical conductivity and the strength of the connection between the nanotube and metal. Our attention was therefore drawn to conductive metal oxides, and specifically, to the intermediate oxides of tungsten, WOx (x = 2-3). With tungsten oxides, when the value of x does not satisfy 3, the growth direction is limited by a planar defect of oxygen layers in the crystal, resulting in spontaneous formation of a rod-shaped structure. We discovered that it is possible to promote epitaxial growth of nanorods with a diameter of 5-20 nm arranged in the axial direction on a tungsten single crystal substrate (**Fig. 1**).

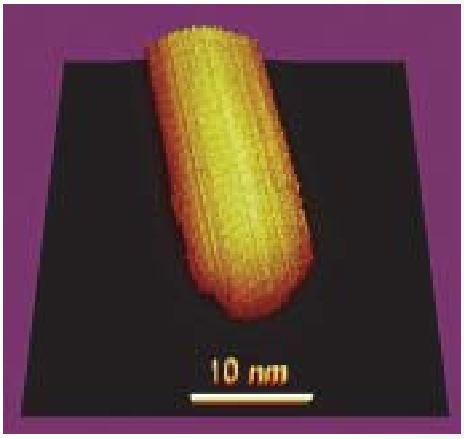

Fig. 1 TEM image of tip of metal oxide nanorod observed by atomic identification electron microscope.

Fig. 2 is an electron microscope image of a nanorod probe actually fabricating using this technique. Here, one nanorod was grown selectively on the tip by performing masking treatment on electrochemically sharpened tungsten. Because these nanorods are electrically conductive, atomic-resolution images can be obtained by using the nanorod probe in scanning tunneling microscope observation (see inserted figure: atomic resolution image of silicon surface). In other words, in MP-STM measurements, we have shown that the position of contact between the nanorod probe and the nanostructure can be measured with atomic accuracy (see **Fig. 3**).

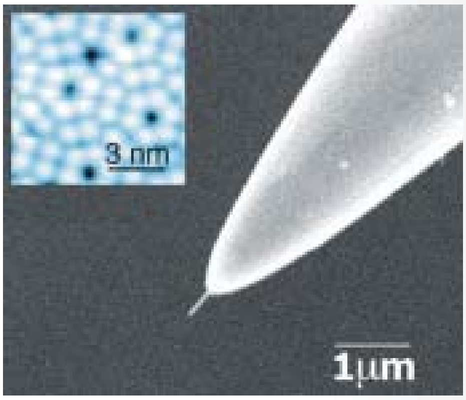

Fig. 2 Nanorod probe and STM image of silicon using the probe.

Conductive metal oxide nanorods are expected to be used in nanocharacterization by MP-STM, for example, in elucidating electron transport phenomena in extremely small structures consisting of a linear arrangement of several molecules or several tens of atoms. As conductive wiring materials which can be self-generated from metal electrodes, they should also play an important role in the nanoelectronics of the future.

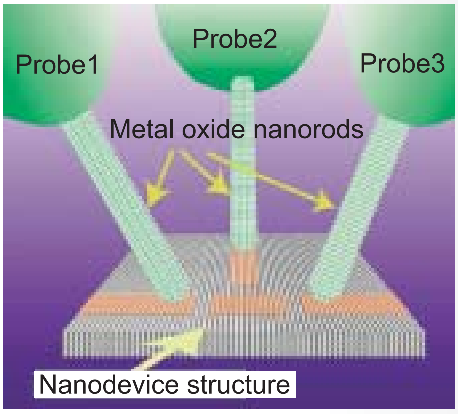

Fig. 3 Schematic diagram showing measurement of electrical conductivity of nanostructure using nanorod probes.

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Second Swiss-Japanese Workshop on Biomaterials

The Second Swiss-Japanese Workshop on Biomaterials was held with joint sponsorship by NIMS and the Swiss National Science Foundation (SNSF) at the NIMS Sengen Site and Tsukuba international convention center, Epochal, over a three-day period from November 5 to 7. Participants included 25 distinguished researchers in the field of biomaterials, with 12 researchers and the Attache for Science and Technology from the Swiss Embassy in Tokyo attending on the Swiss side, and representatives on the Japanese side including NIMS President Teruo Kishi, Dr. Junzo Tanaka, Director of the Biomaterials Center, and Prof. Teruo Okano, Chairman of the Japanese Society for Biomaterials. Although sessions were closed, debate was vigorous and included discussions of personnel exchanges and joint research. The next workshop in this series is to be held in Switzerland in 2005.

Direct measurement of electric resistance of conductive nanowire

—New characterization technique using dual-probe scanning tunneling microscope—

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Tomonobu Nakayama
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Nanomaterials Laboratory(NML)

High integration and high performance in semiconductor devices based on silicon technology are expected to reach their physical limits in the near future. Therefore, direct characterization and evaluation of molecular devices, quantum devices, and other new functional devices, as well as the functions of nano-scale structures used in these devices, is an urgent task for the Electro-nanocharacterization Group in establishing next-generation nano-electronics.

The dual-probe scanning tunneling microscope (DP-STM), which was developed by this Group, is a new characterization instrument that uses two metal probes with sensitivity at the atomic level in the same basic manner as tester electrodes. Because each of the probes has the functions of a scanning tunneling microscope, the DP-STM has the functions of an electrical property measuring device/nano-tester which enables control of the tester electrode position with atomic-level accuracy.

Here, the DP-STM was used to measure the electric resistance of conductive nanowires on a silicon (Si) surface. A single crystal of $ErSi_2$ can be obtained by supplying erbium (Er) atoms on a clean Si(001) substrate. Because the deviation in lattice spacing between the Si crystal and hexagonal $ErSi_2$ crystal is larger in the direction of the c axis than the a axis of the $ErSi_2$ crystal (**Fig. 1**), self-assembled nanowires composed of single crystal $ErSi_2$ can be obtained under proper temperature conditions. The cross-section of these nanowires is 3-10 nm (width) x 1 nm (height), and their maximum length is approximately 1 μm.

In resistance measurements, first, the $ErSi_2$ nanowires on the surface were observed by STM, and one nanowire was selected. The two probes of the DP-STM were then connected to the ends of the selected nanowire (**Fig. 2**). After confirming that the voltage and current between the probes satisfied Ohm's law, resistance R between the probes was measured. Successive measurements of R while narrowing the distance L between the probes showed that R decreases linearly as L is reduced (**Fig. 3**). The resistivity of the nanowire was obtained from the slope of this line. The resistivity of the measured $ErSi_2$ nanowires was approximately one order of magnitude greater than that of bulk $ErSi_2$, which was attributed to surface scattering in the nano-scale conductor.

These measurements, which were first made possible by the DP-STM, truly demonstrate the usefulness of this instrument. In the future, we believe that multi-probe scanning probe type microscopes, as represented by the DP-STM, will become increasingly important in material and element development for realizing nano-electronics.

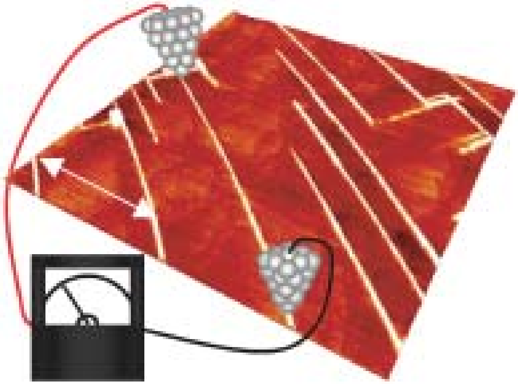

Fig. 2 STM image of $ErSi_2$ nanowire on surface of Si(001).

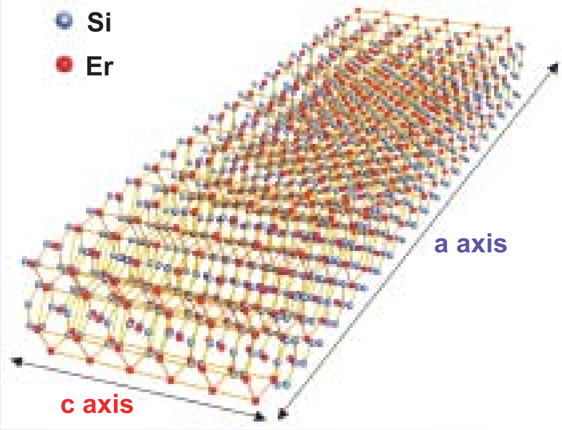

Fig. 1 Model of $ErSi_2$ nanowire structure.

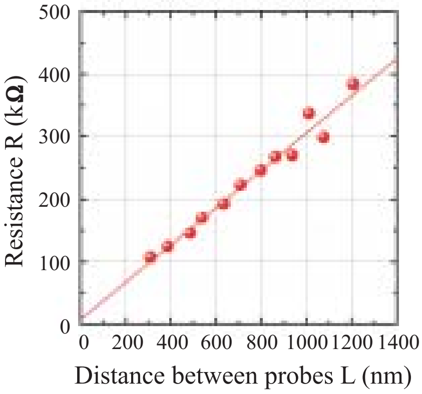

Fig. 3 Relationship between resistance R of nanowire and probe spacing L.

Publication of Datasheets

As part of its commitment to improving the intellectual infrastructure in its Mid-term Plan, NIMS is obtaining atmospheric corrosion property data and development guidelines for low alloy steels, and has published the first two corrosion datasheets in this field, "No. 0 Plan for Prepartion of Corrosion Property Datasheets" and "No. 1 Atmospheric Corrosion Property Datasheet for Fe-Ni and Fe-Cr Binary Alloys."

NIMS also recently published a creep datasheet, "No. 36B Quenched and Tempered Chrome-Molybdenum Steel Plates for Pressure Vessel Use ASTM A542/542M (2.25Cr-1Mo)," which summarizes creep fracture data, including test pieces which fractured a maximum of approximately 180,000 hours (corresponding to 20 years, 6 months).

Development of atomic switch

—Conceptually new device based on atomic mechanics—

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It has been pointed out that the silicon transistor, which is the basis of today's information society, is now approaching its operational limits, both physically and economically. For this reason, the development of new nanodevices is urgently needed.

In contrast to conventional devices, which control electron transfer, we developed a device which operates based on the completely new concept of controlling the movement of atoms. Because the atomic movement required for switching is only 1 nm, performance is superior to that of electronic devices. The development of this atomic switch was made possible using a material called a mixed conductor in one of a pair of opposing electrodes. As a distinctive feature of mixed conductors, when an electric field is impressed, in addition to electrons, the metallic ions which comprise the crystal also move in the crystal.

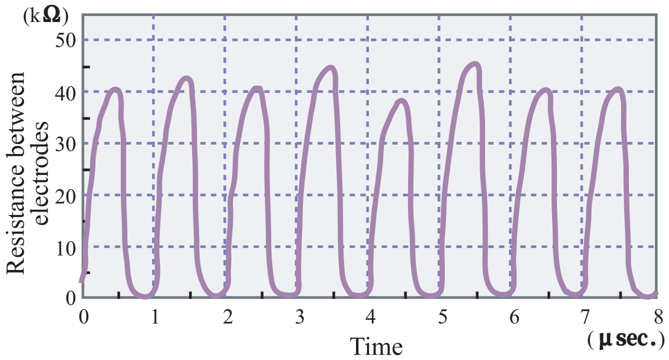

Fig. 1 Results of switching operation at 1 MHz.

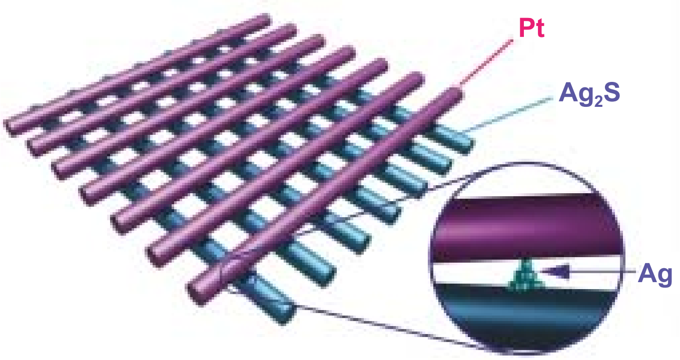

Fig. 2 Atomic switch array with cross-bar structure. Silver atoms are precipitated and form bridges at each intersection point.

We discovered that metallic ions are reduced and precipitate on the surface when tunneling electrons are introduced locally into a mixed conductor, causing the formation of a nanoscale projection. This projection forms a bridge between the opposing electrodes, which are separated by a distance of only 1 nm, turning the switch on. The bridge disappears when a voltage of reversed polarity is impressed, and the switch is turned off. In this research, silver sulfide (Ag_2S) was used as the mixed conductor.

Fig. 1 shows the results of switching operation at 1 MHz using the fabricated switch. Although atoms move at a slower speed than electrons, because the distance between the electrodes, in other words, the distance which the atoms must move in switching, is an extremely short 1 nm, high speed operation on the order of 10 GHz can be expected in the future. We also succeeded in developing a simple method of fabricating atomic switches at the points of intersection between mixed conductor wires and metal wires (**Fig. 2**), and have set goals for reducing manufacturing costs.

Considering these features, atomic switches are expected to be adopted in a diverse range of applications in realizing nonvolatile memories, computational circuits, and new computer architectures. For example, we have already confirmed correct operation of basic logic gates (AND, OR, NOT) fabricated using the developed switch. This means that all types of logic circuits can be fabricated using only atomic switches. We are currently continuing with research aimed at practical application.

Success in Fabrication of new Dendritic Nanostructure by Electron Beam Irradiation

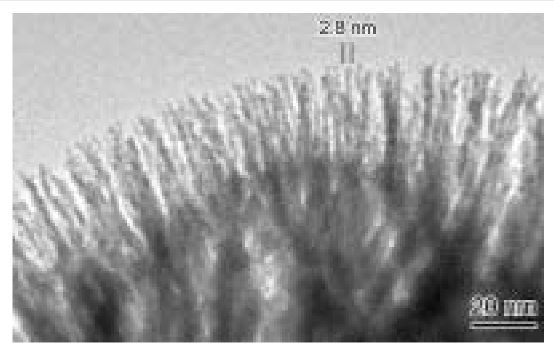

Fig. Dendritic nanostructure.

A Nanomaterials Laboratory (NML) Group headed by Director Kazuo Furuya has succeeded for the first time in the world in fabricating a dendritic nanostructure with a large number branches with diameters of 3 nm by irradiating a convergent electron beam on an organic metal gas. This research makes it possible to inscribe an arbitrary nano-size 2-dimensional pattern on a substrate, and then fabricate a dendritic nanostructure on the pattern. Research and development on fabrication and arrangement of surface-effect devices, sensors, nano-size catalysts and catalyst carrier structures, leading to application in an wide range of fields, including chemistry, biotechnology, and pharmaceuticals, is expected.

Evaluation of TMF properties of Ni-base single crystal superalloy

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Various new heat resistant materials are now under development in the High Temperature Materials 21 Project,* beginning with a Ni-base superalloy for gas turbine stator and rotor blades. As part of this effort, the High Temperature Materials Group has begun evaluating the thermo-mechanical fatigue (TMF) properties of this Ni-base single crystal superalloy which has been developing as TMS superalloy in NIMS (called single crystal superalloy in the following), as described below.

TMF is a type of fatigue which occurs when temperature and strain change simultaneously in combination, and is generally evaluated by the number of cycles to fracture. In this work, TMF properties were obtained using a smooth round-bar specimen of single crystal superalloy and hydraulic servo type fatigue testing machine (MTS Co. type 810 machine,capacity:±5 tons). The temperature cycle was applied to the specimen by induction heating, while the strain cycle was applied with an actuator in the testing machine. To evaluate TMF properties, tests were performed under conditions which simulated TMF in actual turbine blades.

As one example, a test was performed in air with temperature range: 400⇔900℃, total strain range: 1.28%, waveform: triangular wave, frequency: 6 min/cycle, temperature/strain phase: out-of-phase, compressive holding time: 0〜10 hrs. In this case, the maximum compressive strain was generated in the specimen on the high temperature side, while conversely, maximum tensile strain occurred on the low temperature side. Starting and shutdown are comparatively frequent with gas turbines (unit of several hours to 1 week), generating strains in the blades as shown in **Fig. 1**. Moreover, in actual turbines, creep and fatigue act on the blades concurrently. To simulate this condition, compressive strain was held at the maximum temperature for a certain time (see **Fig. 2**).

With this Ni-base superalloy, the number of cycles to TMF fracture is lower with holding time than without holding. We therefore intend to develop a superalloy with the aim of minimizing this decrease in performance.

*For details of this Project, please visit our homepage: http://sakimori.nims.go.jp.

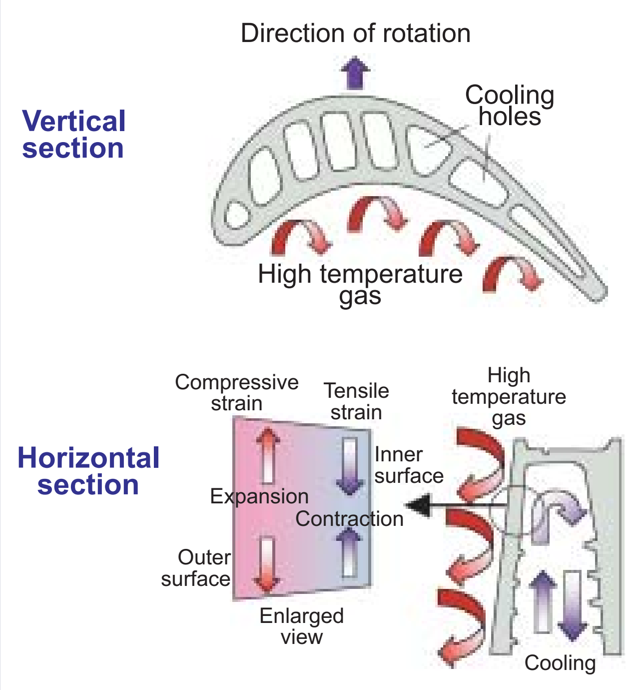

Fig. 1 Strain generated in turbine blade during starting. (Reverse phenomena occur during shutdown.)

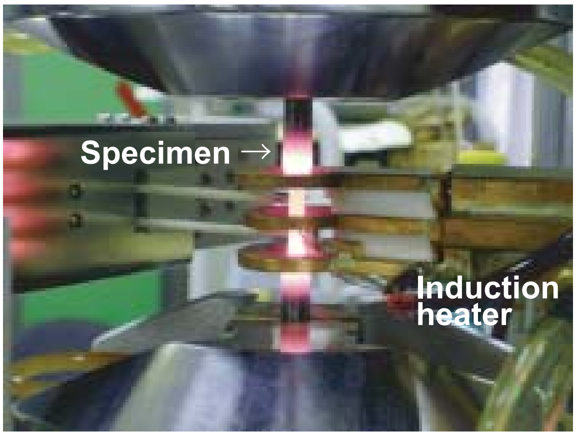

Fig. 2 Appearance of specimen during TMF test.

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Challenges to novel science and technology to establish a new world of nanoelectronics

The third article reports the recent progress in multiple-probe scanning tunneling microscopes (MPSTMs), which we started development several years ago for the first time in the world. MPSTMs are capable of measuring the electrical conductivity of various nanostructures and analyzing the functionality of complex nanostructures directly. The new "nanotesters" will play a key role in the development of novel nanoelectronics, as you will be able to understand from the article.

The fourth article describes the "atomic switch" invented by us. The atomic switch is a completely new type of two-terminal switching device formed by two electrodes; one electrole is made of a solid electrolyte (ion/electron mixed conductor) and the other of a simple metal such as platinum. This structure allows free control of atomic bridging between the two electrodes due to solid electrochemical reaction. The atomic switch possesses a number of outstanding features and attracts great attention as a next-generation nanoelectronic device.

Many tasks remain before nanoelectronics surpasses the present level of today's semiconductor electronics. In our research, we are solving the problems one by one with the goal of realizing a new world of nanoelectronics, which is essential for a highly information-oriented society of tomorrow.

Novel photonic material for arbitrarily shaped two-dimensional waveguides

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Materials Engineering Laboratory (MEL)

Photonic crystals have drawn attention as a photonic material with the potential for realizing microscopic waveguides and high performance light-emitting devices. Photonic crystals are photonic materials which impart periodicity of the wavelength of light to the refractive index, and are fabricated, for example, by arranging rods perpendicular to the surface of a tetragonal lattice at the lattice positions. Because a condition in which light of a certain wavelength cannot propagate is realized, this wavelength region is termed a photonic gap (PG).

The type of waveguide described above is fabricated by partial removal of rods, which means that it is not possible to fabricate waveguides with arbitrary shapes. This is an obstacle to realizing optical integrated circuits.

In contrast, the Intelligent/Smart Materials Group recently discovered a photonic material called uniformly distributed photonic scatterers (UDPS), which possess the photonic gap property and can be used in fabricating arbitrarily shaped two-dimensional waveguides and cavity resonators (which have the potential for realizing high performance in light-emitting devices by strengthening light intensity). Because this is important for practical application, this report will describe the new material.

With UDPS, rods assume a random arrangement when the distance between rods is greater than a certain minimum value (Dmin). A photonic gap is realized when the refractive index and volumetric fraction exceed certain levels. These UDPS have important advantages for actual fabrication. For example, photonic crystals are fabricated using semiconductor technology, but deviations in rod position and fluctuations in rod radii are problems which reduce photonic crystal performance. In contrast, as is obvious from the structure of UDPS, this material is not affected by positional deviations. We have also found that UDPS is resistant to fluctuations in radius.

In fabricating a waveguide, first, the configuration of the waveguide section is decided, and side walls are fenced with rods arranged at fixed intervals to prevent unnecessary light scattering to the sides. Next, the outer region of the waveguide is filled with UDPS. **Fig.** (a) shows a waveguide with a 90 ° bend, and (c) shows an S shape (combination of two 1/4 circles). Measurements of light transmission (white arrow) and the distribution of electrical field intensity (red and yellow indicate strong fields) showed that light transmission is limited to the waveguide and light does not penetrate the surrounding photonic gap UDPS material. Further, (b) and (d) show the calculated results of transmittance on the lines indicated by L in (a) and (c), respectively. The wavelength region shown in gray is a photonic gap, which by nature has transmittance of 0, while the area in the waveguide displays high transmittance.

Future tasks include clarification and experimental study of the mechanism responsible for photonic gaps.

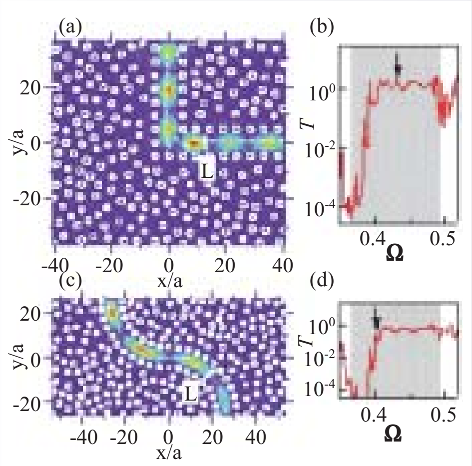

Fig. Waveguide using UDPS and its transmittance.
Main conditions: Dmin = 4a (a: rod radius), refractive index of rods: 3.46, volumetric fraction: 13.8%, fluctuation in radius introduced in range of ±20%, $\Omega = 2\pi a/\lambda$ (λ: wavelength).

Start of Material Design Technology Laboratories— A New Multi-sector Joint Venture

On September 12, 2003, a new venture company named Material Design Technology Co., Ltd. was created through multi-sector cooperation between five organizations, including the independent administrative institutes NIMS and National Institute of Advanced Science and Technology (AIST), Tohoku University, Kyushu University, and InterScience Corporation. One goal of the venture business is to establish a theoretical material design technology in Japan. The company will supply next-generation material design technologies worldwide through sales of databases and computer software, etc. and consulting.

Senior Researcher Furukawa of Advanced Materials Laboratory to Devote Full Efforts to Management of Venture Company

As President of the venture company, OXIDE Corporation, which was established in October 2000, Senior Researcher Yasunori Furukawa of the Advanced Materials Laboratory (AML) has been involved in developing practical applications for research results. Although the company was created only three years ago, its position has steadily improved, including financial and business cooperation with private-sector companies. To achieve further growth in this venture business, Dr. Furukawa resigned from NIMS effective September 30, 2003 and in the future, will devote his full efforts to OXIDE. In August of this year, this company was approved as the 2nd NIMS-supported NIMS Venture Business.

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